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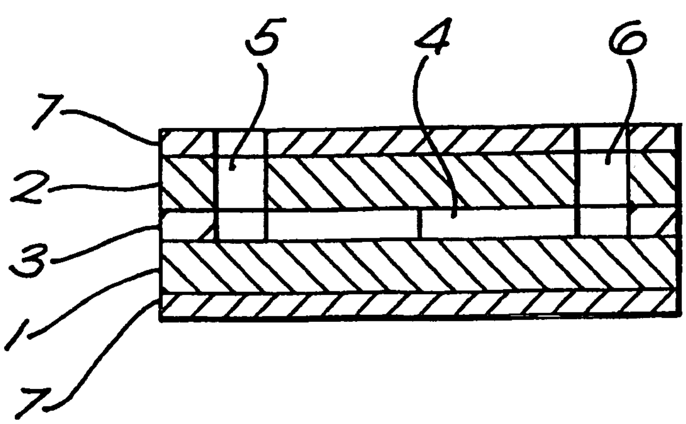
(54) **Cuvette for ir spectroscopy**

(57) The cuvette has inlet and outlet windows (1, 2), at least one sample volume (4) and a spacer 3, and is characterised in that the inlet and the outlet window are designed as wafers (1, 2) of high-resistance silicon, in that the spacer (3) is a layer of silicon dioxide applied to the first wafer (1) and which has a recess (4) surrounded by the layer serving as the sample volume, and in that the second silicon wafer (2) has two openings (5, 6) which emerge in the recess of the silicon dioxide layer, and in that the second silicon wafer (2) is firmly bonded to the silicon dioxide layer.

Openings (5, 6) serve as inlet and outlet for samples undergoing investigation. Wafers (1, 2) may have anti reflex layers (7).



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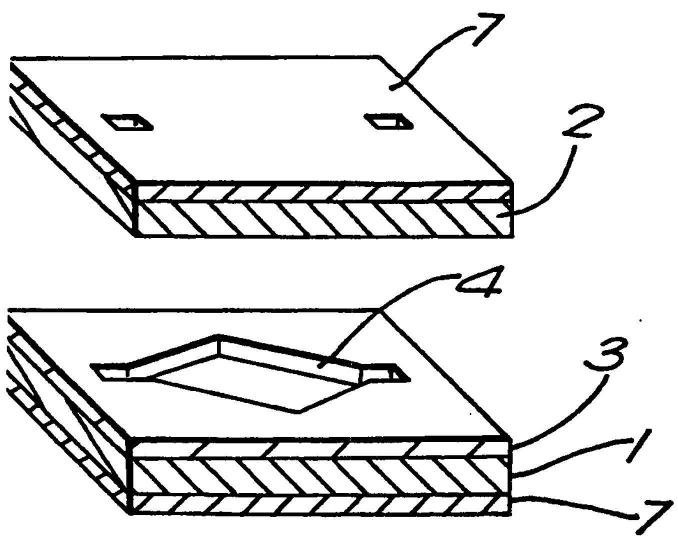
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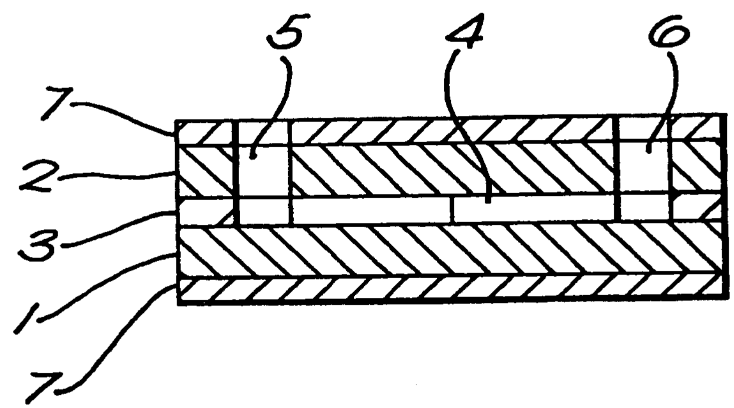
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MICROCUVETTE FOR INFRARED SPECTROSCOPY

This invention relates to a cuvette for use in infrared spectroscopy. Infrared spectroscopy is a simple and quick method, with the assistance of Fourier transformation technology, for characterising the composition of materials.

In this method, molecular vibrations are stimulated in the material to be investigated by means of infrared light at a wave number in the range from $250cm^{-1}$ to $7,000cm^{-1}$ and the absorption maxima are measured.

Conclusions can be drawn regarding the substance to be investigated from the change in position and the height of the absorption maxima by comparing measurements on substances and on known substances of similar composition.

However, the relative data obtained by means of such measurements are very often not adequate. In order to be able to make absolute statements, the absorption cross-sections of the molecular vibrations of interest have to be measured. Series of measurements, in which the absorption on samples of different thickness is determined, are necessary for this.

The higher the absorption for the substance to be investigated, the lower the sample thickness which has to be selected to obtain adequate intensities after transmission of the infrared rays.

Cuvettes having different gap widths, as well as cuvettes with a variable cuvette gap, are offered on the market for the infrared spectroscopy measurements on liquids. However, no cuvettes are available on the market having a gap width lower than 25 µm.

For many substances the absorption by the molecular vibrations of interest is so strong that a signal which

is adequate for evaluation is not transmitted during passage through the cuvette at this gap width.

This disadvantage of the available cuvettes has therefore been hitherto compensated in that the substance to be measured is diluted in a solvent which has only a low absorption in the wave number range of interest.

However, dipole moments and the freedon of movement of the molecular vibrations to be measured may be changed, and hence the position and strength of absorption falsified, due to interactions between the solvent and the substance being investigated. It is therefore desirable to take the measurements on the pure substance instead of on solutions.

Furthermore, the available cuvettes have the disadvantage that the windows which can be penetrated by infrared are either very sensitive to air moisture and/or mechanical stress, and/or are very expensive.

The object of the invention is to provide a cuvette which is not sensitive to air moisture and mechanical stress, and which can be manufactured inexpensively with different and sufficiently low gap width.

According to the invention, there is provided a cuvette for infrared spectroscopy having an inlet and an outlet window, at least one sample volume and a spacer, characterised in that the inlet and the outlet window are designed as wafers of high-resistance silicon, in that the spacer is a layer of silicon dioxide applied to the first wafer and which has a recess surrounded by the layer serving as the sample volume, and in that the second silicon wafer has two openings which emerge in the recess of the silicon dioxide layer, and in that the second silicon wafer is firmly bonded to the silicon dioxide layer.

The cuvette of the invention has an inlet and an outlet window made from high-resistance silicon. This material is one of the most investigated materials because of its use in microelectronics. Good

manufacturing and processing methods are available. The material is not sensitive to moisture and is capable of intense mechanical stress. It has a transmission range for electromagnetic waves having a wave number in the range from $33cm^{-1}$ to $8,300cm^{-1}$ and thus is suitable for infrared spectroscopy.

The spacer between the windows which determines the gap width is a silicon dioxide layer which is applied to one of the silicon wafers, for example by means of epitaxy. The gap width may thus be kept as low as possible and may be varied within a wide range.

The silicon dioxide layer has a recess which serves as the sample volume. The silicon dioxide layer is firmly bonded to the second silicon wafer. Two straight openings in the silicon wafer, which are arranged such that they emerge in the recess of the silicon dioxide layer after fitting together with this layer, serve as inlet and outlet opening for the substance to be investigated. The openings are closed after filling

the sample volume with the substance to be investigated.

Preferably, the silicon dioxide layer has several recesses separate from one another. A cuvette which has several sample volumes which all have exactly the same gap width is thus specified. This cuvette is particularly suitable for simultaneous measurement on different substances.

As already mentioned above, the thickness of the silicon dioxide layer may be varied within a wide range. Preferably, the layer has a thickness of 0.2 to 20 µm. At these layer thicknesses cuvettes are obtained which have a low gap width, such as have not been available hitherto.

Conveniently, the second silicon wafer serving as a window is bonded to the silicon dioxide layer with the aid of silicon wafer bonding. Hence a process which has proved its worth in microstructure technology is used, which leads to a bond which is absolutely tight

and is capable of intense mechanical stress. A cuvette produced in this manner withstands extreme mechanical stresses and is also suitable for liquids having extremely high fluidity.

The inlet and outlet openings may be etched through the silicon wafer to aid manufacture of the cuvette using microstructure technology methods.

The inlet and outlet surfaces of the windows may be vapour-coated with hard anti-reflex layers. The reflection losses of the penetrating infrared ray are thus lowered and scratch resistance of the cuvette increased.

In a preferred embodiment, the cuvette has structured inlet and outlet surfaces for the window. The surfaces are divided into small partial surfaces which are at an angle relative to the total surface. The angle of the partial surfaces is selected so that the penetrating infrared ray meets the partial surfaces at the Brewster angle. Reflection losses may thus be largely avoided

when using a polarised infrared ray. The angle of the partial surfaces is achieved by etching suitably orientated silicon.

Etching silicon at a flank angle of about 70° is required for the infrared light wave numbers used. The partial surfaces may be designed, for example as strips having a width of up to a few 100 µm.

The cuvette can be produced with the aid of processes known to microstructure technology. Silicon wafers serve as starting material here. Several identical cuvettes may be structured and constructed simultaneously on one wafer in one production step.

The essential advantages of the invention consist in also being able to investigate substances using molecular vibrations which lead to very strong absorption without adding solvents. Furthermore, the cuvette of the invention is not sensitive to mechanical stresses and to air moisture. It is also suitable for the use of unknown substances, since the windows are not

attacked by water portions which may be present in such substances. Since silicon has a very low thermal expansion coefficient, the gap width is virtually independent of temperature, so that the cuvette may be used over a wide temperature range.

One exemplary embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawing in which it schematically illustrates the construction of a cuvette of the invention.

A silicon wafer 1 serves as a base for the construction of the cuvette. A conventional silicon wafer having a thickness of 0.5mm may be used by way of example. The construction of several identical cuvettes is carried out in one working step. The identical cuvettes are then separated.

The silicon has doping from 1 to 10 ohm/cm. The transmission range for this material lies at wave numbers from 30 to 8,300cm^{-1}. The penetrability is

about 40%. A change in the absorption strength and position due to interactions of the molecular vibrations with the silicon windows exist for transmission measurements, even for chemical bonds below the detectable level.

A layer of silicon dioxide is applied to the silicon wafer as a spacer 3, for example by means of epitaxy. The layer thickness may easily be varied between approximately 0.2 and 20µm. The silicon dioxide layer contains a recess 4 which serves as the sample volume. The recess is produced, for example by lithography and etching. The shape of the sample volume is adapted to the particular intended use.

A second silicon wafer 2 is firmly joined to the silicon dioxide layer. The bonding may preferably be carried out by means of silicon wafer bonding, or by means of adhesive techniques.

The second silicon layer 2 has two straight openings 5,6 which serve as the inlet and outlet opening for the

substance to be investigated. After bonding with the silicon dioxide layer, the openings 5,6 emerge in the recess 4.

In order to reduce the reflection losses of the infrared ray which enters and leaves, the silicon wafers 1,2 are provided with anti-reflex layers 7.

The length and width of the cuvette may be between a few millimetres and a few centimetres depending on the intended use.

CLAIMS

1. A cuvette for infrared spectroscopy having an inlet and an outlet window, at least one sample volume and a spacer, characterised in that the inlet and the outlet window are designed as wafers of high-resistance silicon, in that the spacer is a layer of silicon dioxide applied to the first wafer and which has a recess surrounded by the layer serving as the sample volume, and in that the second silicon wafer has two openings which emerge in the recess of the silicon dioxide layer, and in that the second silicon wafer is firmly bonded to the silicon dioxide layer.

2. A cuvette according to claim 1, characterised in that the silicon dioxide layer has several recesses separate from one another and the second silicon wafer has two openings per recess which emerge in the recess.

3. A cuvette according to claim 1 or claim 2, characterised in that the silicon dioxide layer has a thickness of 0.2 to 20 μm.

4. A cuvette according to any one of claims 1 to 3, characterised in that the silicon wafer and the silicon dioxide layer are inseparably bonded to one another by means of silicon wafer bonding.

5. A cuvette according to any one of claims 1 to 4, characterised in that the openings in the second silicon wafer are formed with the aid of etching processes.

6. A cuvette according to any one of claims 1 to 5, characterised in that the inlet and the outlet window are vapour-coated with hard anti-reflex layers.

7. A cuvette according to any one of claims 1 to 6, characterised in that the inlet and the outlet window have a microstructure such that the surfaces are divided into partial surfaces which are at an angle so that the incident infrared ray meets them at the Brewster angle.

8. A cuvette according to any one of claims 1 to 7, characterised in that silicon wafers, which are processed using the processes of microstructure technology, serve as starting materials for producing the cuvette, and in that several identical cuvettes may be produced simultaneously in one production step.

9. A cuvette substantially as herein described with reference to the accompanying drawing.

F ents Act 1977
Examiner's report to the Comptroller under Section 17 (The Search Report)

Application number

GB 9223607.4

Relevant Technical fields

(i) UK Cl (Edition K) G1B (BCX)

(ii) Int Cl (Edition 5) G01N

Search Examiner

M R WENDT

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI, CLAIMS

Date of Search

8 DECEMBER 1992

Documents considered relevant following a search in respect of claims 1-9

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
	NONE	

Category	Identity of document and relevant passages	Relevant to claim(s,

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

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